

PATENT COOPERATION TREATY

PCT

NOTIFICATION OF ELECTION

(PCT Rule 61.2)

From the INTERNATIONAL BUREAU

To:

Commissioner
US Department of Commerce
United States Patent and Trademark
Office, PCT
2011 South Clark Place Room
CP2/5C24
Arlington, VA 22202
ETATS-UNIS D'AMERIQUE
in its capacity as elected Office

Date of mailing (day/month/year) 09 April 2001 (09.04.01)	
International application No. PCT/EP00/07174	Applicant's or agent's file reference TS 0854PCT
International filing date (day/month/year) 25 July 2000 (25.07.00)	Priority date (day/month/year) 26 July 1999 (26.07.99)
Applicant VAN DEN BERG, Franciscus, Gondulfus, Antonius et al	

1. The designated Office is hereby notified of its election made:

☒ in the demand filed with the International Preliminary Examining Authority on:
08 February 2001 (08.02.01)

☐ in a notice effecting later election filed with the International Bureau on:

2. The election ☒ was
☐ was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland Facsimile No.: (41-22) 740.14.35	Authorized officer Zakaria EL KHODARY Telephone No.: (41-22) 338.83.38
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(19) World Intellectual Property Organization
International Bureau



(43) International Publication Date
1 February 2001 (01.02.2001)

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(10) International Publication Number
WO 01/07139 A1

(51) International Patent Classification⁷: **B01D 17/04,**
C10G 33/00

STAMPS, Paulus, Antoon [NL/NL]; Badhuisweg 3,
NL-1031 CM Amsterdam (NL).

(21) International Application Number: PCT/EP00/07174

(22) International Filing Date: 25 July 2000 (25.07.2000)

(25) Filing Language: English

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(30) Priority Data:
99305908.8 26 July 1999 (26.07.1999) EP

(71) Applicant (for all designated States except US): SHELL
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(81) Designated States (national): AE, AG, AL, AM, AT, AU,
AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ,
DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR,
HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR,
LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ,
NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM,
TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.

(84) Designated States (regional): ARIPO patent (GH, GM,
KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian
patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European
patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE,
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CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

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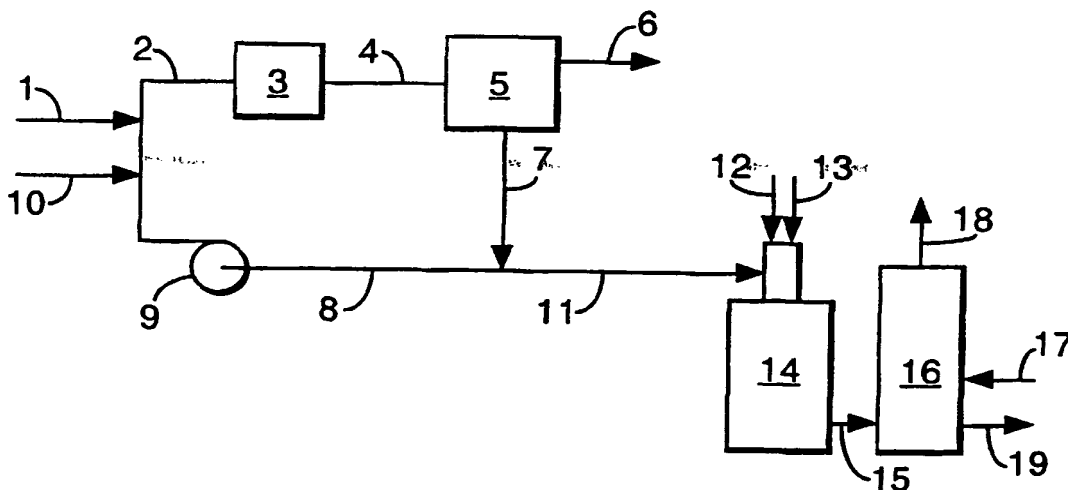
— With international search report.

(72) Inventors; and

(75) Inventors/Applicants (for US only): VAN DEN BERG,
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For two-letter codes and other abbreviations, refer to the "Guid-
ance Notes on Codes and Abbreviations" appearing at the begin-
ning of each regular issue of the PCT Gazette.

(54) Title: BREAKING OF OIL/WATER EMULSION



(57) Abstract: Process for separating an emulsion of a bituminous oil and water into a liquid water phase and a liquid bituminous oil phase, wherein the following steps are performed: (a) raising the temperature of the bituminous oil/water emulsion having a temperature of below 100 °C to a temperature of above 140 °C, and (b) performing a phase separation wherein a water phase and oil phase is obtained, wherein the heating of the emulsion in step (a) is effected by first mixing part of oil phase obtained in step (b) having a temperature of above 140 °C with the bituminous oil/water emulsion and subsequently raising the temperature of the resulting mixture to a temperature of above 140 °C by making use of indirect heat exchange means.

WO 01/07139 A1

INTERNATIONAL SEARCH REPORT

Intern: 31 Application No

PCT/EP 00/07174

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 B01D17/04 C10G33/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 B01D C10G E21B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP 0 790 292 A (MITSUBISHI HEAVY IND LTD) 20 August 1997 (1997-08-20)	1-9
X	page 3, line 8-45	10
A	US 5 441 548 A (BRANDL ADRIAN ET AL) 15 August 1995 (1995-08-15)	1-9
X	cited in the application column 4, line 36 -column 5, line 43	10
A	WO 95 34522 A (MOBIL OIL CORP) 21 December 1995 (1995-12-21)	1-10
	page 6, line 9-22; figure 1	
A	US 5 882 506 A (OHSOL ERNEST O ET AL) 16 March 1999 (1999-03-16)	1-10
	column 3, line 1-31	

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

* Special categories of cited documents :

- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier document but published on or after the international filing date
- "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- "O" document referring to an oral disclosure, use, exhibition or other means
- "P" document published prior to the international filing date but later than the priority date claimed

- "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- "&" document member of the same patent family

Date of the actual completion of the international search

2 November 2000

Date of mailing of the international search report

10/11/2000

Name and mailing address of the ISA

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Authorized officer

Persichini, C

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/EP 00/07174

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP 0790292 A	20-08-1997	JP 9217072 A US 6033448 A JP 9316470 A	19-08-1997 07-03-2000 09-12-1997
US 5441548 A	15-08-1995	DE 4032045 A ZA 9108022 A	23-04-1992 29-07-1992
WO 9534522 A	21-12-1995	CA 2184191 A US 5885424 A	21-12-1995 23-03-1999
US 5882506 A	16-03-1999	AU 1416899 A EP 1032621 A WO 9925795 A	07-06-1999 06-09-2000 27-05-1999

PATENT COOPERATION TREATY

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INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

REC'D 07 NOV 2001

Applicant's or agent's file reference TS 0854 PCT		FOR FURTHER ACTION See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)	
International application No. PCT/EP00/07174	International filing date (day/month/year) 25/07/2000	Priority date (day/month/year) 26/07/1999	
International Patent Classification (IPC) or national classification and IPC B01D17/04			
Applicant SHELL INTERNATIONALE RESEARCH ... et al.			

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.


2. This REPORT consists of a total of 5 sheets, including this cover sheet.

☒ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of 5 sheets.

3. This report contains indications relating to the following items:

- I ☒ Basis of the report
- II ☐ Priority
- III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV ☐ Lack of unity of invention
- V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI ☐ Certain documents cited
- VII ☒ Certain defects in the international application
- VIII ☐ Certain observations on the international application

Date of submission of the demand 08/02/2001	Date of completion of this report 02.11.2001
Name and mailing address of the international preliminary examining authority:  European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Authorized officer Persichini, C Telephone No. +49 89 2399 8617



**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT**

International application No. PCT/EP00/07174

I. Basis of the report

1. With regard to the **elements** of the international application (*Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17)*):

Description, pages:

1,2,4-9 as originally filed

3,3a,10 as received on 12/07/2001 with letter of 12/07/2001

Claims, No.:

1-9 as received on 12/07/2001 with letter of 12/07/2001

Drawings, sheets:

1/1 as originally filed

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/EP00/07174

- ☐ the description, pages:
☐ the claims, Nos.:
☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)

6. Additional observations, if necessary:

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes:	Claims	1-9
	No:	Claims	
Inventive step (IS)	Yes:	Claims	1-9
	No:	Claims	
Industrial applicability (IA)	Yes:	Claims	1-9
	No:	Claims	

2. Citations and explanations
see separate sheet

VII. Certain defects in the international application

The following defects in the form or contents of the international application have been noted:
see separate sheet

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT - SEPARATE SHEET**

International application No. PCT/EP00/07174

- (1) US-A-5 441 548
- (2) EP-A-0 790 292
- (3) US-A-5 882 506
- (4) WO-A-95 34 522

Re Item V

1. Documents (1) and (2), are both considered to represent the closet prior art. The subject-matter of claim 1 differs therefrom in that the emulsion of oil and water to be separated is heated in two steps: first by mixing the emulsion feed with a part of the oil phase obtained in the separation process; thereafter by an indirect heat exchanger.

The temperature rise resulting from the first step lowers the viscosity of the emulsion. According to the applicants, this results in a lower pressure drop in the subsequent heat exchanger so that smaller and more simple pumps, smaller heat exchange means and process equipment designed for lower pressure levels can be used.

The process of claim 1 is neither disclosed nor suggested in the present prior art documents.

Documents (3) and (4) which also concern the separation of oil and water emulsions, disclose the addition of a diluent in order to achieve a viscosity which is favourable for subsequent separation steps. Following to the separation steps, the diluent is removed and recycled. The addition of the diluent is, however, not combined with a heating step.

2. Claims 2 to 8 are dependent on claim 1. Claim 9, as well, can be considered to be dependent on claim 1 as it comprises all the features thereof.

Re Item VII

It is not clear whether the object on which the application is based, is achieved, ie whether the advantages (lower viscosity) resulting from the recirculation of a part of the product prevail the disadvantages (reduced yield as a part of the liquid bituminous oil

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT - SEPARATE SHEET**

International application No. PCT/EP00/07174

phase already separated from the aqueous phase is remixed to the feed; increased amount of liquid which has to be pumped through the heat exchanger).

C L A I M S

1. Process for separating an emulsion of a bituminous oil and water into a liquid water phase and a liquid bituminous oil phase, wherein the following steps are performed:

5 (a) raising the temperature of the bituminous oil/water emulsion having a temperature of below 100 °C to a temperature of above 140 °C, and

(b) performing a phase separation wherein a water phase and oil phase is obtained,

10 wherein the heating of the emulsion in step (a) is effected by first mixing part of oil phase obtained in step (b) having a temperature of above 140 °C with the bituminous oil/water emulsion and subsequently raising the temperature of the resulting mixture to a temperature
15 of above 140 °C by making use of indirect heat exchange means.

2. Process according to claim 1, wherein in step (a) the temperature is raised to a value of between 140-200 °C.

3. Process according to claim 2, wherein in step (a) the
20 temperature is raised to a value of between 160-200 °C.

4. Process according to claim 3, wherein the temperature of the resulting mixture is raised from a value of between 120-150 °C to a value of between 160-180 °C by making use of the indirect heat exchange means.

25 5. Process according to any one of claims 1-4, wherein the pressure in step (b) is sufficiently high in order to obtain both phases in the liquid state.

6. Process according to claim 5, wherein in step (b) the liquid water phase has a pH of below 7.

30 7. Process according to claim 6, wherein the pH of the liquid water phase is between 4 and 6.

Amended
1990/07/14

8. Process according to any one of claims claim 1-7,
wherein the starting emulsion has a water content of
between 10-40% by weight, a surfactants content of
between 0.01-5% by weight and an oil content of between
5 60-85% by weight, wherein the oil alone has a viscosity
of above 305 Pa.s at 20 °C.

9. Process according to claim 8, wherein the emulsion is
an ORIMULSION.

10 10. Use of the oil phase obtained in the process
according to any one of claims 1-9 having a temperature
of above 140 °C as feedstock in a gasification process.

Example 2

To one weight part of a typical ORIMULSION (ORIMULSION is a trade name of Intevep S.A. describing an emulsion of a bituminous oil and water and their preparation is described in US-A-4795478) an amount of sulphuric acid was added in order that the pH of the resulting water phase had a pH of 5 (80 mg sulphuric acid per kg of emulsion). The water content of the emulsion was 30% by weight. Phase separation was performed at a temperature of 180 °C and at a pressure of 10 bar. The phases remained in the liquid phase during phase separation. A water phase was obtained as the top phase. In Table 1 some more information of the ORIMULSION used and the resulting phase separation is presented.

Comparative Example

Example 2 was repeated except that no acid was added. The pH of the resulting water phase was 7.9. See also Table 1.

Table 1

	Orimulsion	Example 2 Bituminous oil phase	Comparative experiment Bituminous oil phase
Calcium (ppmw)	34	12	52
Magnesium (ppmw)	46	5	46
oil in water phase after phase separation (wt%)		0.1	0.3

order to perform the desired raise in temperature or, as is the case in US-A-5441548, more than one heat exchanger will have to be used in series. The high pressure needed is disadvantageous because special pumps must be used. Furthermore the heat exchanger and the process equipment downstream of the heat exchanger, like for example the gravity-type emulsion separator, must be designed for this higher pressure level for obvious safety reasons. The present invention provides a process which can be operated at a lower pressure having all the obvious advantages in view of the above.

This object is achieved by the following process. Process for separating an emulsion of a bituminous oil and water into a liquid water phase and a liquid bituminous oil phase, wherein the following steps are performed:

(a) raising the temperature of the bituminous oil/water emulsion having a temperature of below 100 °C to a temperature of above 140 °C, and
(b) performing a phase separation wherein a water phase and oil phase is obtained, wherein the heating of the emulsion in step (a) is effected by first mixing part of oil phase obtained in step (b) having a temperature of above 140 °C with the bituminous oil/water emulsion and subsequently raising the temperature of the resulting mixture to a temperature of above 140 °C by making use of indirect heat exchange means.

It has been found that by mixing the emulsion feed with part of the bituminous oil phase obtained in the phase separation of the emulsion the temperature can be sufficiently raised in order to lower the viscosity of the mixture entering the heat exchanger means. This results in that a lower pressure drop in the heat exchanger has to be overcome enabling a lower inlet

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INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference TS 0854PCT	FOR FURTHER ACTION see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.	
International application No. PCT/EP 00/ 07174	International filing date (day/month/year) 25/07/2000	(Earliest) Priority Date (day/month/year) 26/07/1999
Applicant SHELL INTERNATIONALE RESEARCH MAATSCHAPPIJ B.V.		

This International Search Report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This International Search Report consists of a total of 2 sheets.

☒ It is also accompanied by a copy of each prior art document cited in this report.

1. Basis of the report

- a. With regard to the **language**, the international search was carried out on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.

☐ the international search was carried out on the basis of a translation of the international application furnished to this Authority (Rule 23.1(b)).

- b. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international search was carried out on the basis of the sequence listing :

☐ contained in the international application in written form.

☐ filed together with the international application in computer readable form.

☐ furnished subsequently to this Authority in written form.

☐ furnished subsequently to this Authority in computer readable form.

☐ the statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.

☐ the statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished

2. ☐ **Certain claims were found unsearchable** (See Box I).

3. ☐ **Unity of invention is lacking** (see Box II).

4. With regard to the **title**,

☒ the text is approved as submitted by the applicant.

☐ the text has been established by this Authority to read as follows:

5. With regard to the **abstract**,

☒ the text is approved as submitted by the applicant.

☐ the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.

6. The figure of the **drawings** to be published with the abstract is Figure No.

☒ as suggested by the applicant.

☐ because the applicant failed to suggest a figure.

☐ because this figure better characterizes the invention.

1
☐ None of the figures.

INTERNATIONAL SEARCH REPORT

International Application No

PCT/EP 00/07174

A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 B01D17/04 C10G33/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 B01D C10G E21B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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	column 3, line 1-31 -----	

☐ Further documents are listed in the continuation of box C.

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- "A" document defining the general state of the art which is not considered to be of particular relevance
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- "P" document published prior to the international filing date but later than the priority date claimed

- "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- "&" document member of the same patent family

Date of the actual completion of the international search

2 November 2000

Date of mailing of the international search report

10/11/2000

Name and mailing address of the ISA

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Persichini, C

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/EP 00/07174

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP 0790292 A	20-08-1997	JP 9217072 A US 6033448 A JP 9316470 A	19-08-1997 07-03-2000 09-12-1997
US 5441548 A	15-08-1995	DE 4032045 A ZA 9108022 A	23-04-1992 29-07-1992
WO 9534522 A	21-12-1995	CA 2184191 A US 5885424 A	21-12-1995 23-03-1999
US 5882506 A	16-03-1999	AU 1416899 A EP 1032621 A WO 9925795 A	07-06-1999 06-09-2000 27-05-1999